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EUROPEAN PATENT APPLICATION

② Application number: 82304843.4

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C 11 D 3/32

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③ Priority: 16.09.81 GB 8127967

⑦ Applicant: UNILEVER PLC, Unilever House Blackfriars P
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⑧ Designated Contracting States: GB

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⑤ Antifoam compositions.

⑤ Anti-foam compositions suitable for use in detergent
powders and liquids are formed from polysiloxane liquids and
selected hydrophobic particles. The particles in question are
alkylene dialkylamides, polyvalent metal salts of alkyl phos-
phoric acids or polyvalent alkali metal salts of carboxylic acids,
or precursors of any of these.

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ANTI-FOAM COMPOSITIONS

This invention relates to anti-foam compositions.

Anti-foam compositions are widely used in industry, especially in the detergents, textile and paper industries. The chemical nature of the anti-foam materials which have
5 been suggested in the past is very diverse, but this invention is concerned with certain mixtures of polysiloxanes with defined particulate solids.

According to the broadest aspect of this invention there is provided an anti-foam composition comprising the
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Anti-foam compositions are widely used in industry, especially in the detergents, textile and paper industries. The chemical nature of the anti-foam materials which have
5 been suggested in the past is very diverse, but this invention is concerned with certain mixtures of polysiloxanes with defined particulate solids.

According to the broadest aspect of this invention there is provided an anti-foam composition comprising the
10 combination of a hydrophobic liquid and a particulate solid, characterised in that the hydrophobic liquid is a polysiloxane and the particulate solid comprises an

α, ω -dialkylamide alkane, a polyvalent metal salt of an alkyl phosphoric acid or polyvalent alkali metal salt of an alkyl carboxylic acid, or a precursor of such a salt.

Specific mixtures of polysiloxanes and particulate solids have been disclosed as anti-foam compositions. For example, British Patent No 1,407,997 discloses mixtures of polysiloxanes and hydrophobic silicas and US Patent No 3,235,509 discloses mixtures of polysiloxanes with hydrophobed aluminium oxide.

The present invention provides a further choice of anti-foam compositions which are useful for a variety of applications, but which are especially useful for producing low foaming soap and detergent compositions.

According to a narrower aspect of the invention, therefore, there is provided a detergent composition characterised by containing an anti-foam composition comprising the combination of a hydrophobic liquid and a particulate solid, wherein the hydrophobic liquid is a polysiloxane and the particulate solid comprises an α, ω -dialkylamide alkane, a polyvalent metal salt of an alkyl phosphoric acid or a polyvalent metal salt of an alkyl carboxylic acid, or a precursor of such a salt. By a precursor, in this context, is meant a free acid or a monovalent salt which will react with polyvalent metal ion in solution to form the desired species.

It is preferred that the particulate solid comprises an α, ω -dialkylamide alkane or a polyvalent metal salt of an alkyl phosphoric acid.

When the anti-foam composition is incorporated into a fabric washing powder and when the polysiloxane is mobile, then it will be desirable to protect the anti-foam composition, otherwise the mobile liquid will migrate away from the particulate solid, leading to deactivation of the foam-inhibiting properties. Protection techniques which are known in the detergents art are appropriate here. For example, the anti-foam composition can be encapsulated in a

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water-soluble or water-dispersible medium such as gelatin or polyethylene glycol, or it may be granulated on a spherical or cylindrical core of sucrose or gelatinised starch and subsequently coated with, for example, paraffin wax.

5 The polysiloxanes used in the invention are generally alkyl polysiloxanes. These materials are well known to industrial chemists and are available in a wide range of molecular weights, the higher molecular weight materials, 10 as is usually the case with polymers, tending to be more viscous. Polysiloxanes are available from various suppliers, for example Dow Corning Corporation of Illinois, USA, and Rhône-Poulenc Chemie, France.

15 The anti-foam composition will normally be present in detergent compositions in accordance with the invention in amounts of from 0.1 to 2%, preferably 0.1 to 1%.

It will be understood that this invention is concerned with an anti-foam composition and consequently no attempt is being made in this specification to describe all 20 possible uses to which the composition can be put. However the compositions are especially effective when incorporated into a liquid detergent and we prefer that it should be applied to liquid detergent compositions of both the structured and unstructured types. When the composition 25 is to be used in a detergent composition then it is self-evident that the usual detergent composition components can be incorporated provided that they have no adverse reaction with the components of the anti-foam composition. For example, anionic surfactants such as 30 alkylbenzene sulphonates, primary and secondary alkyl sulphates, secondary alkane sulphonates, soaps and olefine sulphonates can be used. Nonionic surfactants, either alone or in combination with anionic surfactants can also be used, the preferred nonionic surfactants being C₇ to 35 C₂₄ primary or secondary alcohols ethoxylated with from 1 to 25 moles of ethylene oxide per mole of alcohol.

Typical amounts of surfactant are from 3 to 25% by weight when only one species is present, and from 1 to 16% by weight when more than one is present.

Builders may be present in amounts of from 5 to 50% by weight. Typical of the inorganic builders are sodium tripolyphosphate, sodium pyrophosphate and sodium orthophosphate, sodium carbonate and the crystalline and amorphous forms of aluminosilicates. Organic builders such as sodium nitrilotriacetate, sodium citrate, sodium carboxymethyloxysuccinate, and the host of other materials have been suggested as phosphate replacers are also appropriate.

Other components which may be present are sodium silicate as a corrosion inhibitor and powder structurant, oxygen bleaches such as sodium perborate and sodium percarbonate, bleach precursors such as tetraacetylene diamine, fluorescers, antiredeposition agents and anti-ashing agents, suds-suppressing agents other than the anti-foam compositions of this invention, and moisture.

The optional components listed above are mainly suitable for compositions in powdered (granular) form although some are also suitable for incorporation into liquids. When the compositions are in liquid form, optional components particularly appropriate to this form include electrolytes, hydrotropes, opacifiers, stabilising polymers, organic solvents and colourants.

Generally speaking, the anti-foam composition will contain from 1 to 40% by weight of the particulate solid.

The invention is illustrated in the following examples:-

Example 1

A soap-based fabric washing composition was prepared by standard spray-cooling and dry-dosing techniques to the following formulation.

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		<u>Parts by weight</u>
	Sodium soap of C ₁₂₋₂₂ fatty acids	41.3
	Coconut oil ethanolamide	2.5
	Sodium tripolyphosphate	10.0
5	Sodium silicate (Na ₂ O:SiO ₂ , 2.5:1)	8.0
	Sodium carbonate	2.9
	Magnesium silicate	0.5
	Sodium carboxymethyl cellulose	0.3
	Sodium perborate	20.0
10	Fluorescer, antioxidant and water	10.0

Two solutions of the above powder were prepared. The first, Solution A was a solution of the powder alone in demineralised water at a concentration of 5 grams per litre, and the second, Solution B was a similar solution in which an anti-foam composition in accordance with the invention had been dispersed by ultrasonic agitation at a concentration of 0.2 grams per litre. The anti-foam composition was a 20:80 mixture by weight of the calcium salt of a mixture of phosphoric acids sold under the registered trade mark "Alf 5" with a polysiloxane oil (Dow Corning's 200/1000cs). "Alf 5" contains 63% of the C₁₆₋₁₈ monoester of phosphoric acid and 7% of the diester.

The foam produced by both solutions at a series of temperatures was assessed using a Ross-Miles foam meter. The results are shown in Table 1.

Table 1

	<u>Temperature (°C)</u>	<u>Foam Volume (mls)</u>	
		<u>Solution A</u>	<u>Solution B</u>
		(control)	(the invention)
30	30	250	100
	40	280	115
	50	255	135
	60	255	170
35	70	260	195

It can be seen that the foam volume generated in the apparatus by Solution A, the control is higher than that of Solution B containing the anti-foam of the invention at all temperatures.

Example 2

A structured liquid washing composition having the following formulation was prepared.

	<u>% by weight</u>
Sodium soap	33.6
¹ Alkyl sodium benzene sulphonate	4.2
² Ethoxylated alcohol	10.0
Triethanolamine	10.0
Ethanol	10.0
Glycerol	3.5
Boric Acid	1.5
Water and minor components	balance to 100.0

¹ Marlon AS3 (registered Trade Mark)

² Dobanol 91-6 (registered Trade Mark) supplied by Shell Chemicals Limited.

The pH of the composition was adjusted to 9.8.

This composition, and two similar ones in which 0.1% and 0.2% by weight of the water were replaced, respectively with 0.1% and 0.2% by weight of a 1:9 mixture of methylene distearamide and a liquid polysiloxane having a viscosity of 1000 cs were subjected to test in the following manner.

200 grams of the liquid were dosed into a miele 436 (registered Trade Mark) washing machine containing a full load of clean washing. The washing was then washed using the high temperature cycle. During the process the machine consumed 16 litres of water of 9° French hardness.

The suds generated during the process was measured against an arbitrary scale applied to the machine. The results are shown in Table 2.

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Table 2

		Foam Height (arbitrary)		
	Time (mins)	No Anti-foam	0.1% Anti-foam	0.2% Anti-foam
5	0	2.8	0	0
	5	3.5	0	0
	10	3.5	0	0
	15	1.0	0	0
	20	0.5	0	0
10	25	0.6	0	0
	30	3.0	0.2	0.1
	35	3.0	1.0	0.5
	40	3.5	3.5	2.0
	45	4.0	3.5	2.0

It can be seen from the above figures that the defined combinations of methylene distearamide are effective anti-foams for the composition described, and we believe that these results are typical of those which are obtained from other anti-foam combinations claimed herein.

Example 3


A liquid detergent composition stabilised with polyacrylic acid was prepared to the following formulation:

		% by weight
	Sodium dodecyl benzene sulphonate	7.5
	³ Ethoxylated alcohol	2.5
25	Sodium tripolyphosphate	20.5
	Sodium tetraborate	4.6
	Glycerol	3.0
	⁴ Polyacrylic acid	0.5
	Water and minor components	balance to 100.0
30	³ Synperonic 7EO (registered Trade Mark), a primary C ₁₁₋₁₅ 7EO ethoxylate supplied by Imperial Chemical Industries Limited.	
	⁴ Carbopol 941 (registered Trade Mark) supplied by B F Goodrich Company.	
35	The pH of the composition was adjusted to 8.5.	

This composition, and two similar ones in which 1.0% and 2.0% by weight of the water were replaced with 1.0% and 2.0% by weight respectively of a 1:9 mixture of ethylene distearamide and a polysiloxane having an average viscosity of 1000 cs to 60 000 cs were subjected to test in the following manner.

100 grams of the liquid were dosed into a Miele 429 (registered Trade Mark) washing machine containing a full load of clean washing. The washing was then washed using the high temperature cycle. During the process the machine consumed 3.5 gallons of water of 24° French hardness. The suds generated during the process was measured as described in Example 2. The results are shown in Table 3.

Table 3

		<u>Foam Height (arbitrary)</u>		
	<u>Time (mins)</u>	<u>No Anti-foam</u>	<u>1% Anti-foam</u>	<u>2% Anti-foam</u>
	0	0	0	0
	5	Overflow	0.3	0.3
20	10		1.8	0.3
	15		4.3	0.3
	20		6.0	0.5
	25		8.0	1.8
	30		10.0	3.5
25	35		10.5	6.5
	40		11.0	6.7
	45		11.5	8.0

It can be seen from these figures that an antifoam in accordance with the invention is effective in the defined liquid composition, although as the inherent foam generating power of the surfactants used in this instance are greater than in Example 2, somewhat larger amounts of anti-foam are required.

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CLAIMS

1. An anti-foam composition comprising the combination of a hydrophobic liquid and a particulate solid, characterised in that the hydrophobic liquid is a polysiloxane and the particulate solid comprises an
- 5 α, ω dialkylamide alkane, a polyvalent metal salt of an alkyl phosphoric acid or polyvalent alkali metal salt of an alkyl carboxylic acid.
2. An anti-foam composition according to claim 1 characterised in that the particulate solid comprises an
- 10 α, ω dialkylamide alkane or a polyvalent metal salt of an alkyl phosphoric acid.
3. An anti-foam composition according to claim 1 or claim 2 characterised in that at least the hydrophobic liquid is enclosed in a water-soluble or water-dispersible
- 15 envelope.
4. An anti-foam composition according to any one of the preceding claims characterised by a content of the particulate solid of from 1 to 40% by weight.
5. A detergent composition characterised by comprising an
- 20 anti-foam composition according to claim 3 or claim 4.
6. A detergent composition according to claim 5 in the form of a liquid.
7. A detergent composition according to claim 6 in the form of a structured liquid.

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EUROPEAN SEARCH REPORT

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Application number

EP 82 30 4843

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl. 3)
Y	FR-A-2 338 990 (UNILEVER N.V.) * Claim 1 *	1,2	C 11 D 3/00 C 11 D 1/82 C 11 D 3/32
Y	FR-A-2 338 991 (UNILEVER N.V.) * Claims 1, 2 *	1,2	
D,A	GB-A-1 407 997 (PROCTER & GAMBLE CO.) * Claims 1, 2 *		
A	DE-A-2 909 757 (CIBA-GEIGY AG) * Claims 1, 7 *		
The present search report has been drawn up for all claims			TECHNICAL FIELDS SEARCHED (Int. Cl. 3)
			C 11 D 1/00 C 11 D 3/00
Place of search BERLIN		Date of completion of the search 27-07-1983	Examiner SCHULTZE D

CATEGORY OF CITED DOCUMENTS

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